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Mechanical integrity of glass ceramic restorations on Morse taper implant-abutment system

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It has been a great honor to have been able to learn from professors and colleagues at Federal University of Santa Catarina (UFSC) in Brazil. In my life, success has never been a gradual continual slope upwards. Rather, it has always had many difficulties and challenges that wind their way through life that have ultimately led to success. Whether it was the clinic ceiling catching on fire, multiple university strikes, power outages, or even just catching a bus to Lagoa, my time in Brazil was no different. I came to UFSC with what I believed to be a well-thought out plan that quickly showed major, fundamental flaws. One-of-which being my proficiency in Portuguese.

Even though I was not able to enroll in majority of my classes the first four months of my study, my new UFSC colleagues and professors were willing to try and help me feel comfortable amidst the daily difficulties. With a week to spare before classes started the next semester, Professor Izabel Santos Almeida and Magini agreed to accept me into the dental implantology Master's Program the week before classes started.

Thus, I began an intense pursuit of research within my allotted timeframe of January 2015-December 2015. In these moments, I was fortunate enough to have been supported by many professors and colleagues within the clinic, classroom, and the emerging research proposal. I will be forever grateful to the professor's collaboration and contribution among CEPID, CERMAT and VITROCER at the UFSC. Without this willingness for productivity and acknowledgement of common goals and outcomes, I am unsure if I would have been able to overcome my research criteria and timeline.

RESUMO

O principal objetivo deste estudo foi avaliar a integridade mecânica após fadiga de coroas de silicato de lítio reforçadas com zircônia cimentadas e conectadas a sistemas de implantes do tipo cone Morse. Quarenta implantes de titânio foram inseridos em bases de poliacetal simulando osso de suporte. Os pilares protéticos correspondentes foram conectados aos implantes usando um torque de 20 N.cm controlado por um torquímetro digital portátil. Estruturas protéticas unitárias foram projetadas por computador para produzir coroas vitrocerâmicas de dissilicato de lítio (LD) ou silicato de lítio e zircônio (LZS) por termo-pressão. Após cimentação e conexão das coroas, os grupos implante-pilar-coroa foram submetidos a testes de fadiga (n = 10) sob uma carga de 200 N em 500.000 ciclos a 5 Hz imersos em solução eletrolítica a 37 °C. Depois dos ensaios de fadiga, as coroas de cada grupo foram removidas para avaliar valores de torque de remoção do pilar protético ao implante (*n* = 7). Os grupos de coroa-implante-pilar restantes foram seccionadas transversalmente em 90° em relação ao plano da conexão pilar-implante para a inspeção de fraturas e *microgaps* por microscopia eletrônica de varredura. Grupos controle foram imersos na solução eletrolítica mas não foram submetidos aos testes de fadiga. Após imersão em solução e sem efeito de fadiga, os valores de torque de remoção dos pilares para os grupos LD e LZS estavam em 18,014 ± 1,634 N.cm e 18,214 ± 0,813 N.cm, respectivamente. No entanto, os valores de torque de remoção diminuíram significativamente em ambos os grupos, registrando valores de 12,8 ± 1,6 N.cm e 14,9 ± 1,08 N.cm, para o grupo LD e o grupo LZS, respectivamente ($p < 0,05$). Não houve diferença significativa nos valores de torque de remoção entre os dois grupos não submetidos à fadiga assim como entre os dois grupos teste (após fadiga). A análise microscópica revelou que o tamanho dos *microgaps* (0,9 ± 0,3 µm) encontrado em conexões implante-pilar aumentou significativamente (4,2 ± 0,9 µm) para os grupos submetidos a testes de fadiga ($p<0,05$). Além disso, foram detectadas micro-trincas nas interfaces entre coroa-adesivo e adesivo-pilar em ambos os sistemas após testes de fadiga. As coroas vitrocerâmicas à base de silicato de zircônio e lítio cimentadas e aparafusadas aos implantes dentários resistiram aos testes de fadiga simulando um período de seis meses de mastigação. No entanto, os resultados confirmaram uma diminuição do torque nos pilares com o aumento dos *microgaps* das conexões pilar-

implante e a presença de micro-trincas nas interfaces coroa-adesivo como consequência do estímulo mecânico dinâmico.
Palavras-chave: silicatos de lítio e zircônio; LZS; ensaios de fadiga; implantes dentários; implante-pilar.

ABSTRACT

The main aim of this study was to assess the mechanical integrity of zirconium- lithium silicate crowns cement- and screw- retained to a Morse taper titanium implant-abutment system after fatigue. Forty titanium implants were placed in poliacetal to mimic bone support. Abutments were torqued to the implants on 20 N.cm using a digital handheld torque meter. Each implant-abutment received a unit maxillary premolar crown that was computer-designed and hot-pressed. Crowns were divided into two groups: A) lithium disilicate (LD); or B) zirconium-lithium silicate glass-ceramic (LZS). Implant-abutment-crown groups were submitted to mechanical cycling tests (n = 10) on 200 N at 5 Hz for 500,000 cycles in a Ringer's electrolytic solution (37 °C). After fatigue tests, crowns from each group were removed to evaluate removal torque values on abutment to implant. The remaining crown-implant-abutment assemblies were cross-sectioned at 90° to the implant-abutment joint for inspection of cracks and the microgap by scanning electron microscopy. After immersion in Ringer's solutions, removal torque values without effect of fatigue recorded for LD group were at 18.0 ± 1.6 N.cm while values at 18.2 ± 0.8 N.cm were recorded for LZS group. However, removal torque values after fatigue tests significantly decreased down to 12.8 ± 1.6 N.cm for LD while LZS group revealed values at 14.9 ± 1.1 N.cm for LZS ($p < 0.05$). There was no significant difference in torque values between the test groups after fatigue. Microscopic analyses revealed that the microgap size found at implant-abutment connections at about 0.9 ± 0.3 μm significantly increased up to 4.2 ± 0.9 μm for the groups subjected to fatigue tests. Also, cracks at the crown-adhesive or at adhesive-abutment interfaces were detected at both systems after fatigue tests. Zirconium-lithium silicate glass-ceramic crowns cement- and screw-retained to dental implants were mechanically successful under fatigue testing in an electrolyte solution. However, the findings confirmed a loosening of mechanical integrity of the crown-adhesive-abutment interfaces as well as decrease in removal torque values on abutment to implant joints after fatigue.

Keywords: Lithium and zirconium silicates glass-ceramics, LZS, Fatigue Tests, Dental Implants.

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LIST OF ABREVIATIONS

BISGMA - Bisphenol A Glycidyl Methacrylate
BISEMA – Ethoxylated Bisphenol-A-dimethacrylate
CAD-CAM - Computer-Aided Design & Computer-Aided Manufacturing
CEPID - Center for Research on Dental Implants
CERMAT - Ceramic & Composite Materials Research Laboratories
DEJ – Dentin-enamel Junction
FGM - Functionally Graded Materials
LD – Lithium disilicate glass ceramic
LZS - lithium-zirconium silicate glass ceramic
PEEK - Polyether-ether-ketone
PFM - Porcelain fused to Metal
PTFE – Polytetrafluorethylene
SEM- Scanning Electron Microscopy
TEGDMA - Triethylene Glycol Dimethacrylate
UDMA – Urethane Dimethacrylate
VITROCER - Vitreous Materials Laboratory
YTZP - Yttria stabilized Tetragonal Zirconia Polycrystal

1. INTRODUCTION

Within the last 50 years, dental implants have become the standard of care for edentulous spaces within the modern dentition. The purpose behind dental implants is to anchor a functional, dental prosthetic crown to allow for proper mastication, phonetic, and esthetic outcomes. The fundamental problem associated with implant dentistry is the lack of a periodontal ligament, which anatomically allows for stable, long-term flexural patterns between the supporting bone tissues and the natural dentition. The lack of an periodontal ligament adds additional flexural stress within the implant-abutment system likely transferring increased loading forces throughout the entire implant system and the supporting soft and hard tissues [1].

Flexural overload of titanium-bone interface will cause chronic long-term resorption or loss of osseointegration during life of implant. Thus, that has led to a major concern of fatigue strength of implant supported all-ceramic prostheses [2–5]. Long-term mechanical cycling of glass ceramics can cause crack propagation leading to failure of the prosthetic crown [6–8]. High stress magnitude from occlusal loading can lead to cementation failure of crown to implant-abutment system [5,9]. Moreover, overload transmission from abutment to implant results in micromechanical movements of the contacting surfaces within the microgap connection space between the abutment and implant [10]. Micro-movements lead to a decrease of friction on the connecting structural materials that cause a loosening of the mechanical integrity of the implant system.

Additionally, the concept of cementation or bonding an all-ceramic crown to an titanium implant-abutment system has little definitive research to validate the long-term efficacy of such prosthetic options [9-12]. Recently, the improvement of dental ceramics such as zirconia and glass ceramics have reduced crack propagations associated with shearing forces which has significantly improved the structural integrity of ceramics to be considered as a viable alternative for dentistry [6,13] . However, the mechanical integrity of these modern glass ceramics has not been extensively investigated relative to implant-supported loads.

The main aim of this study was to investigate the mechanical integrity of implant supported cemented glass ceramic prosthetic groups through mechanical cycling testing, torque values, and microscopic evaluation. The null hypothesis was that the mechanical performance of dental implant-supported prostheses represented by the microstructural integrity of crown-adhesive-abutment interfaces and by torque value maintenance of abutments to Morse taper implant systems was not affected by mechanical cycling.

The present master thesis was organized and elaborated in sections. An initial literature review detailing importance aspects, namely: dental implants, dental implant supported prosthetics, distribution of load through the implant system, aesthetic prosthetics, dental cements, bonding in implant dentistry and emerging novel materials in dentistry.

The following sections describe the materials and methods used in this study highlighting the preparation and experiental analysis of two dental implant-supported all ceramic prosthetic groups: lithium disilicate (LD) and zirconium lithium silicate (LZS) glass ceramics. The findings are elucidated within the final sections: results, discussion and conclusions. The main outcome of this study supported a significant difference in removal torque values recorded on abutments submitted or not to fatigue tests. SEM images further illuminated micro-cracks at ceramic-adhesive-abutment interfaces as well as microgap widening between implant and abutment. These findings were reported and submitted for publishing in an international scientific index journal.

2. LITERATURE REVIEW

2.1 *Dental Implants*

The marked increase in demand for dental implants has stimulated the development of novel designs and structural materials in the field of implant dentistry. The concept behind dental implant functionality is anchorage of a prefabricated implant screw design that is embedded into a host bone tissue site (Fig. 2.1). The foreign implant must be biocompatible to allow for long-term osseointegration within the osseous hard tissue for anchorage and support of the prosthetic crown.

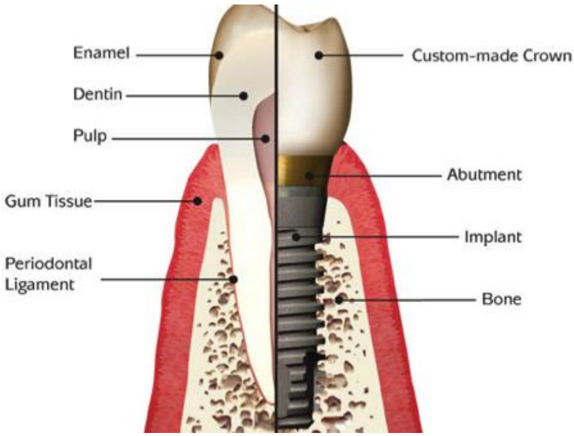


Figure 2.1. Schematic illustration of structures of natural dentition and implant-supported prosthesis (http://consideringdentalimplants.co.uk/index.html).

The lack of a periodontal ligament (as seen in Fig. 2.1.) found within edentulous regions that have been restored using osseointegrated dental implant systems is a current limitation found in implant dentistry. The loss of osseointegration is a significant factor behind the development of novel emerging materials that are being incorporated into the dental implant system. Concerning implant-bone interaction, the traditional use of commercially pure titanium and titanium alloys has shown a highly biocompatibility and corrosion resistance due to the titanium oxide layer formed on the surface. That allows for long-term success in traditional dental implant osseointegration [14,15]. However, there remains a relevant concern of bone loss saucerization at the dental implant platform level, which has led to various novel designs of implant-abutment connections [16,17].

Historically, as noted in Figure 2.2, dental implants to abutment connections have used an external or internal hexagonal connection or a more recent internal Morse taper (conical connection). Different abutment (I) and implant (II) connections as well as the implant-abutment assembly (IV and V) can be seen in Figure 2.2. Of these modern designs, internal implant-abutment connection designs have revealed several advantages including aesthetic and biomechanical behavior [1-5, 18-22]. Also, internal conical connection dental implant-abutment design has shown to reduce bone loss associated at the bone-level connection.

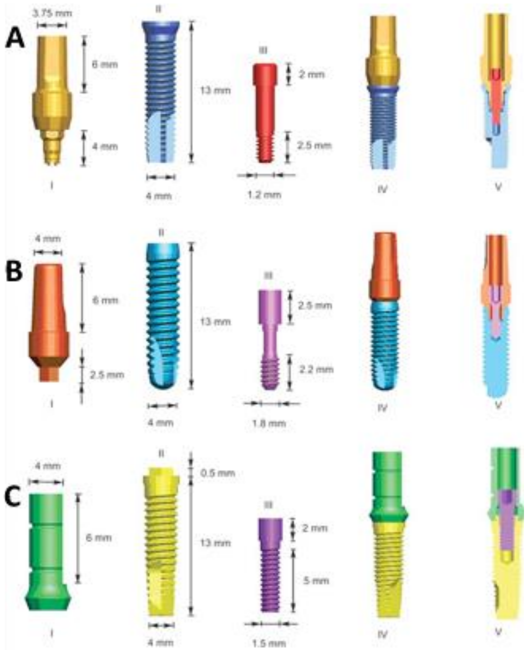


Figure 2.2. Comparison of Dental Implant Systems: A) Internal Hexagon Connection B) Internal Conical Morse taper Connection C) External Hexagonal Connection. I) abutment; II) implant; III) abutment screw; IV) implant-abutment assembly; V) cross-sectioned implant-abutment assembly [18].

Additionally, conical connection designs have shown increased stability and parallelism throughout the joint space [17]. This design feature has reduced the microgap size existing in the connection and the torque failure associated with other types of implant-abutment connections and enhancing the long-term prognosis of dental implant systems. Commercially pure (CP) titanium is frequently used to fabricate implant fixtures while the abutment, torqued on implant, can be produced from titanium alloys; or else from ceramic materials such as yttria-stabilized zirconia.

2.2 *Implant Supported Prosthesis*

The purpose behind dental implants is to anchor a functional, dental prosthetic crown to allow for proper mastication, phonetics, and esthetic outcomes [14,15,19]. Over the last 50 years, modern dentistry has incorporated ceramic materials to mimic the color and appearance of the natural dentition over more traditional metal-based dental restorations [19,22]. Nowadays, ceramic materials are used to synthesize metal-free (e.g. zirconia-porcelain systems) and metal-ceramic (e.g. feldspar-based ceramic fused on metallic materials) crowns (Fig. 2.3). Screw or cement-retained crowns are two forms of retaining the implant-abutment prosthetic [11]. Screw-retained crowns are commonly selected over cement-retained crowns due to their ability to be easily retrieved and reduced risk of peri-implantitis associated with excess cement [14]. However, in esthetic zones where resorbed boney ridges limit space for screw access holes cement retained crowns are typically recommended. An additional advantage to cement retained crowns is a lower frequency of fracture strength or failure rates associated with crack propagation around access hole in screw-retained porcelain crowns [3-5].

In many clinical cases where esthetic outcomes are a major factor in treatment planning the metal abutment substructure limited an adequately translucent crown. Additionally, considering mechanical behavior, more fracture prone feldspar-based porcelain required a metallic coping or substructure to support an esthetically appealing prosthetic traditionally known as a porcelain fused to metal (PFM) crown [6-8,19]. These metal-based materials had the natural ability to be molded into form, withstand the masticatory forces, and the acidic nature of internal oral environment with highly predictable outcomes, yet lacking an esthetic, natural appearance.

Thus, the initial revelation for ceramics used in modern dentistry showed significantly improved esthetic appearances, yet higher failure rates associated with fractures, crack propagation and poor cement and/or bonded retention [6]. Within the last thirty years, improvement of the ceramic performance, bond strength, esthetic outcome has allowed for full coverage porcelain restorations to become a highly predictable, esthetically appealing prosthetic option in modern dentistry [6–8].

Figure 2.3. Traditional Porcelain fused to metal (left) and all ceramic prosthetic (right).

The major flaw found with metallic dental implant systems is a higher associated modulus of elasticity (also known as elastic modulus or Young's modulus) that ultimately results in overload on the bone tissues supporting the implant system. Within current implant systems, some emerging novel materials such as polyether-ether-ketone (PEEK) and resin filled composite has shown limited success. However this fundamental flexural variance has not able to be successfully overcome [3,21]. In many clinical cases where esthetic outcomes are a major factor in treatment planning the metal abutment substructure limits an adequately translucent crown. The overall long-term success of the titanium implant-abutment system is still considered the current standard of care and ultimately the prosthetic design has to be altered in order to adapt to this implant system.

Regarding esthetic crowns, this increased elastic modulus historically has led to a major flaw between fatigue strength of the esthetic ceramic crown supported by dental implants. The traditional metal abutment and the more traditional ceramic crowns required a metallic coping or substructure to support an esthetic prosthetic. Recently, the improvement of dental ceramics including zirconia and glass ceramics have reduced crack propagations associated with shearing forces which has significantly improved the structural integrity of ceramics to be considered as a viable alternative for implant dentistry. This allows the dental clinician to have the highest esthetic outcome with similar long-term prosthetic outcomes found in metal supported crowns.

2.3 *Materials for esthetic single unit prosthesis*

Traditional dental porcelains types incorporated tectosilicate mineral feldspar ($KAlSi_3O_8$), quartz (SiO_2) and kaolin ($Al_2O_3.2SiO_2.2H_2O$). Feldspar-based ceramics have shown excellent aesthetic properties, but these ceramics were never recommended in the higher load bearing posterior dentition due to flexural strength at 100-160 MPa and low fracture toughness [6-8,13,22].

Lithium silicate glass-ceramic contains approximately 65% volume fraction of lithium disilicate, 34% volume fraction of residual glass and 1% volume fraction of porosity after heat treatment [40]. The glass matrix is derived from a multi-component system, formulated from SiO_2-Li_2O-K_2O-ZnO-Al_2O_3-La_2O_3-P_2O_5 glass system [36-40]. Previous studies have reported the manufacturing of glass-ceramic materials belonging to the Li_2O-ZrO_2-SiO_2-Al_2O_3 (LZSA) or Li_2O-ZrO_2-SiO_2 (LZS) glass-ceramic systems for different applications. LZSA glass-ceramics containing about 55% volume of glass powder can be obtained using injection molding sintering ranging from 570 up to 850°C and then crystallizing of the glass powder compacts [23-26]. The main crystalline phases formed for LZSA glass-ceramics were β-spodumene, zirconium silicate and lithium metasilicate. For a given sintering temperature and time in the 650–850°C temperature range, Vickers hardness values ranged between 3.3 ± 0.2 and 9.6 ± 1.0 GPa while bending strength values was between 100 ± 12 and 197 ± 19 MPa [41-43]. Regarding LZS glass- ceramics, $Li_2Si_2O_5$ and $ZrSiO_4$ were the resulting main crystalline phases within the microstructure after sintering until 850 or 900°C. LZS glass-ceramics revealed a Vickers hardness at 8 ± 0.5 GPa, an elastic modulus at 115 ± 0.42 GPa, bending strength of 214 ± 20 MPa and fracture toughness at 3.65 ± 0.21 $MPa.m^{1/2}$ [23-26, 41-43]. Such experimental values described here are quite proper on single-unit fixed dental prostheses for anterior rehabilitation.

With the recent introduction of Vita Suprinity PC (Vita Zahnfabrik, Germany), zirconium lithium silicate reinforced glass ceramics are now available as alternative to lithium disilicate glass ceramics. It should be highlighted that the zirconium lithium silicate (LZS) material used in this study, has been produced and studied at the glass-ceramic research laboratory (VITROCER) at the Dept. of Mechanical Engineering at Federal University of Santa Catarina).

Nevertheless, it should be emphasized that the integrity of the monolithic zirconia or lithium disilicate glass-ceramic crown structure can be readily compromised by processing defects such as pores and cracks as well as during finishing by grit blasting or polishing. Thus, the brittle mechanical behavior of the glass-ceramics remains a concern regarding catastrophic fracture or abrupt stress distribution across the structural materials used in implant-supported prostheses. A high concentration of stress at prosthetic structural materials can increase the risk of brittle fractures at the crown-adhesive-abutment interfaces [27,36-43]. Recently, efforts have been made to avoid failures in the manufacturing of dental ceramics and also to create ceramic-specific mechanisms to prevent crack propagation [8,11,28,29]. Also, the addition of fillers in the microstructure of glass-ceramic including crystalline particles or short fibers has enhanced the strength and long-term reliability of ceramic prostheses [8,19,22].

The emergence of polycrystalline ceramics such as alumina and zirconia has greatly enhanced flexural strength values and allowed for an esthetic, non-metal, alternative for posterior dental prosthetics. Based upon sintering temperatures, unalloyed zirconia has three crystallographic forms: monoclinic (room temperature to 1170 °C, tetragonal from 1170 °C to 2370 °C and cubic 2370 °C to the melting point. The addition of stabilizing oxides such as yttria (Y_2O_3) enhances the major phase stabilization of the zirconia crystalline form known as tetragonal zirconia polycrystal (TZP) [27,39-40]. That tetragonal phase is the most stable, highest strength crystalline phase. Under load bearing stimuli, the yttria stabilized,Y-TZP, transforms from the partially stable to more stable monoclinic phase [39-40]. Additionally, low temperature degradation of the crystalline form from the major metastable tetragonal phase to the minor monoclinic phase in the presence of water has the primary drawback of yttria-stabilized zirconia and long-term efficacy in biomedical applications [36].

2.4 *Cements and bonding principles*

The classical need for bonding agents or cements in dentistry has been based upon luting a man-made restorative material to tooth structure. Using modern techniques to reduce excess cement leakage during fixation of crown to abutment has reduced incidence of peri-implantitis cases. This act of luting two materials using micromechanical interlocking is commonly referred in dentistry as cementing [32]. Also, bonding is considered a physical and chemical union of two objects using chemical bonding and physical interlocking. The development of polymerization based resin cements have allowed for increased properties of translucency, low solubility, and less incidence of excess cement inflammatory reactions [32]. Based upon their chemical form of polymerization, resin cements can be classified as light-cured, self-cured, and dual-cured. This implies that these resin compounds can be initiated for polymerization and cured via light polymerization, chemical auto-polymerization, or both. Additionally, more recent improvements in acid-etch pre-treatment of bonding surface have improved overall bond strength. Total-etch (etch and rinse) versus self-etch (acid etching and primer contained within resin cement) surface treatment systems have allowed for two different clinical techniques of resin cements application and surface-treatment [32,33].

When cementing or bonding a restorative material to a metal abutment, basic surface treatment has been recommended to improve bonding strength. Various studies have shown air-abrading the titanium metal surface with 50 μm sand-grit to significantly increase bond strength [30]. Using an acid-etch to increase surface roughness has also shown to be an effective pre-surface treatment. However, it is common practice to acid etch the restorative dental ceramic with a 5-10% HF acid, yet few studies have elucidated the effectiveness of HF acid surface treatment of the metal abutment surface and bond strength [30,31].

Tsuchimoto et al [12] showed the effect of surface pretreatment bond strength on acid etched titanium plates with resin cements. Of the two acids tested HCl and H_3PO_4, no significant difference was noted in immediate tensile bond strength. On the other hand, thermal cycling significantly decreased bond strength. It was also noted that the chemical anaylsis of the titanium surface post acid-etch treatment showed a reduction of carbon from the HCl, and an increase in phosphate groups in the H_3PO_4 groups. Additionally, after thermal cylic testing, a significant difference in strictly adhesive failure (H_3PO_4) and mixed failure (HCl) was observed. Novel procedures such as acid etching of crown and sand blasting of abutment have enhanced bond strength and improved long-term success of cemented implant-supported crowns [30,31].

The main components of bonding agent are the resin matrix and silica or silicate micro- and nano-fillers. This composition can be varied depending upon use and proprietary blend of each brand. These components allow for the most successful physicochemical bond between restorative material and tooth abutment structure. The silicate fillers can be altered in size and include nano-fillers until micro sized fillers that will alter viscosity, bond strength, and shrinkage rate of bonding agent. The resin matrix is commonly composed of methacrylate-based monomers such as BISGMA, UDMA, BISEMA and TEGDMA. Currently all bonding agents require light curing to allow for initial free radical propagation to occur and terminate the chemical reaction [8,12,32,33].

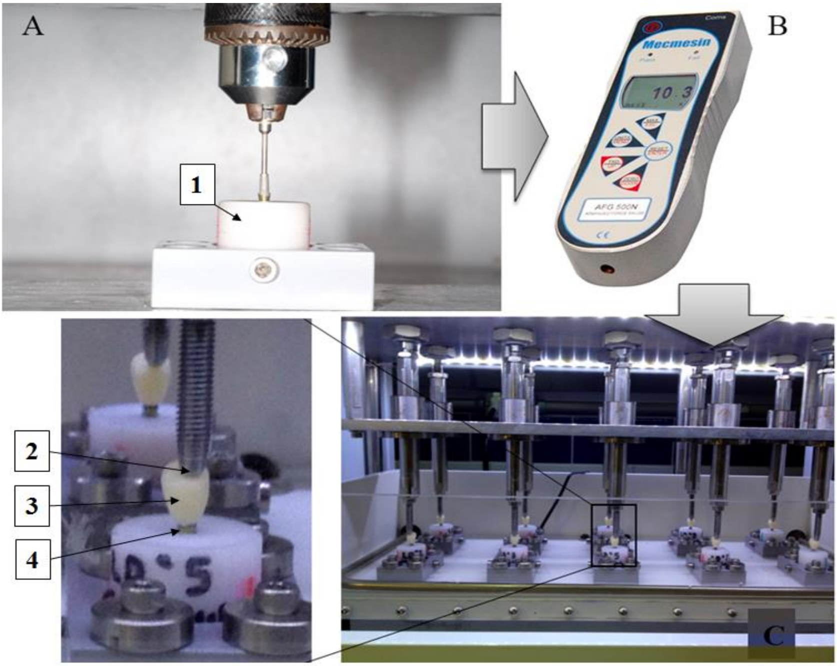
3. MATERIALS AND METHODS

3.1 *Surface Treatment of Titanium Abutment Surface*

Forty titanium alloy (Ti6Al4V) abutments were embedded in a condensation silicone bed exposing only the cervical abutment surface for surface treatment. The bonded surface area of the titanium abutment was airborne-particle abraded with 50-µm aluminum oxide (Al_2O_3) at 0.2 MPa 10 mm away from nozzle for 10 s. Then, abraded surfaces were acid etched with 10% HF solution for 1 min. The titanium surfaces were rinsed in sterile water under ultrasonic bath for 5 min. All surfaces were dried with oil-free air at room temperature.

3.2 *Connection of the implant-abutment assemblies*

Forty Morse taper dental implants (S.I.N. Implants, São Paulo, Brazil) made of commercially pure (cp) titanium grade IV with 3.5 mm diameter and 10 mm length were placed into poliacetal holding devices by torqueing at 80 N.cm using a handheld digital torque meter (Mecmesin Advanced Force Gauge, United Kingdom), as seen in Figure 3.1A and 3.1B. The poliacetal holding devices were produced by CAD-CAM to ensure an accurate implant placement. Morse taper abutments (S.I.N Implants, São Paulo, Brazil) made of Ti6Al4V alloys were tightened to implants on 20 N.cm (Fig. 3.1A and B) by using the digital torquemeter coupled to a metallic holding device in order to avoid oblique loads [44,45].



1. Poliacetal electrolytic holding device; 2. Counterbody; 3. Glass ceramic crown; 4. Morse taper abutment.

Figure 3.1. Torque of conical connection titanium abutment to implant using handheld digital torque meter. A) Abutment on torque application. B) Digital handheld torque meter.

Helping reduce abutment removal and altering torque values, the abutment screw access channel was filled with polytetrafluoroethylene (PTFE) tape after initial placement of abutment to implant. Then, the abutment screw access hole was covered using self-curing luting composite (Multilink Automix, Ivoclar Vivadent, USA).

3.3. *Processing and cementation of the glass ceramic crowns*

A digital impression of the titanium abutment post-surface treatment was carried out by computer-assisted design (CAD) to produce a maxillary first premolar prosthetic crown to mimic ideal dimensions. Crowns were designed with a screw access hole in the central fossa of the premolar occlusal surface in order to access the abutment for removal torque values before and after fatigue testing. The premolar design mimicked a maxillary first premolar of the adult dentition. The lingual cusp tip of crown was designed to be only area in contact (2 x 2 mm) during fatigue test to mimic oblique loads during dynamic fatigue tests. These crowns were fabricated using 3-D printing machine of investment dies. Following heat pressed investment of crowns, all crowns were hand trimmed and polished. Prosthetic crown single units were divided into two groups considering the ceramic systems: (A) Lithium disilicate glass-ceramic (LD) (IPS e.Max press, Liechtenstein) and (B) Zirconium- lithium silicates glass ceramic (LZS) produced in the Laboratory of Glass-Ceramic Materials (VITROCER, Federal University of Santa Catarina, Florianópolis, Brazil) illustrated in Table 3.1. Details on composition, mechanical properties and clinical application of the materials used in this work are shown in Table 3.2.

Table 3.1. Study group sample size and testing environment.

<table>
<tr><th>Groups</th><th>Removal Torque measurement (n)</th><th>Microscopic inspection (n)</th><th>Test Conditions</th></tr>
<tr><td>Group A1: LD prosthesis-Implant-Abutment</td><td rowspan="4">7</td><td rowspan="4">3</td><td rowspan="2">Immersion in Artificial Saliva (AS) for 72 h without fatigue testing</td></tr>
<tr><td>Group B1: LZS prosthesis-Implant-Abutment</td></tr>
<tr><td>Group A2: LD prosthesis-Implant-Abutment</td><td rowspan="2">Immersion in Artificial Saliva (AS) on Fatigue Testing for 72h</td></tr>
<tr><td>Group B2: LZS prosthesis-Implant-Abutment</td></tr>
</table>

Prosthetic crown surfaces were ultrasonically cleaned immersed in 90% alcohol and dried at room temperature. Then, the inner surfaces of lithium silicate porcelain crowns were etched with 5% hydrofluoric acid (IPS Ceramic Etching Gel, Ivoclar Vivadent, Liechenstein) for 20 s and the rinsed in distilled water for 60 s before drying using oil-free air at room temperature. Each crown was cemented to the corresponding abutment using a self-curing luting resin composite (Multilink Abutment, Ivoclar Vivadent, USA). In order for the polymerization to continue the chemical curing reaction unhindered, margins of the crowns were covered with an air-blocking gel (Liquid Strip, Ivoclar Vivadent, Liechenstein). Following polymerization, the remaining excess luting composite was meticulously removed using hand scalers without intraoral limitations.

Table 3.2. Data on the test materials considering standard composition, elastic modulus, strength and clinical applications.

Materials (brand)	Composition (% weight)	Elastic Modulus (GPa)	Strength (MPa)	Clinical application
Lithium Disilicate (IPS e.Max press, Ivoclar Vivadent)	SiO_2 (57-80); Li_2O (11-19); K_2O (0-13); P_2O_5 (0-11); ZrO_2 (0-8); ZnO (0-8); Others	95	Flexural: 400	Single-tooth restorations, bridges for the anterior and premolar region and implant superstructures.
Lithium-Zirconium silicate glass-ceramic (LZS), VITROCER - UFSC)	Li_2O (9.56) ;ZrO_2 (22.36): SiO_2 (68.08) *Samples sintered and crystallized at 900°C for 120 min.	*115.7 ± 1.1	*Flexural: 191 ± 12	-
Commercially pure titanium grade IV (S.I.N.® implants)	Ti (0.3); Fe (0.2); O (0.015); H (0.05); N (0.08); C (0.355)	104.1-130	Tensile: 550	Dental implants; metal-ceramic prostheses infrastructures.
Adhesive agent (Multilink Automix, Ivoclar Vivadent)	Dimethacrylates and HEMA (32.5); Barium glass filter (Ba-Al-Fluoro-Silicate) (37.4); Ytterbium trifluoride (23); Silica (5.4); Others.	≥ 3 (Self-curing: 4.51; Dual-curing: 6.19).	Flexural: ≥ 50 (Self-curing: 98; Dual-curing: 114).	Cementation of indirect restorations
Ti6Al4V alloy	90% Titanium 6% Aluminum 4% Vanadium	150	Tensile: 900-950	Abutment; metal-ceramic prostheses infrastructure

3.4 *Mechanical cycling tests and removal torque measurement*

Fatigue tests on twenty implant-abutment-premolar prosthetic designs (*n* = 10) were conducted on both groups (LZS or LD) under 200 N for 500,000 cycles at 2 Hz (72 h of immersion) immersed in a hypotonic Ringer's solution using an artificial chewing device (Biopdi, São Paulo, Brazil) (Fig. 3.2). That simulated 6 months of chewing cycles in accordance with previous studies [44,45]. The remaining twenty specimens (control group, *n* = 10) were submersed in same hypotonic Ringer's solution for 72 h without fatigue testing (Table 3.1). Followed the fatigue testing, seven samples from each group had composite covering the abutment access hole removed and removal torque value of the abutment screw recorded using the same handheld digital torque meter (Mecmesin Advanced Force Gauge, United Kingdom) stabilized in holding device.

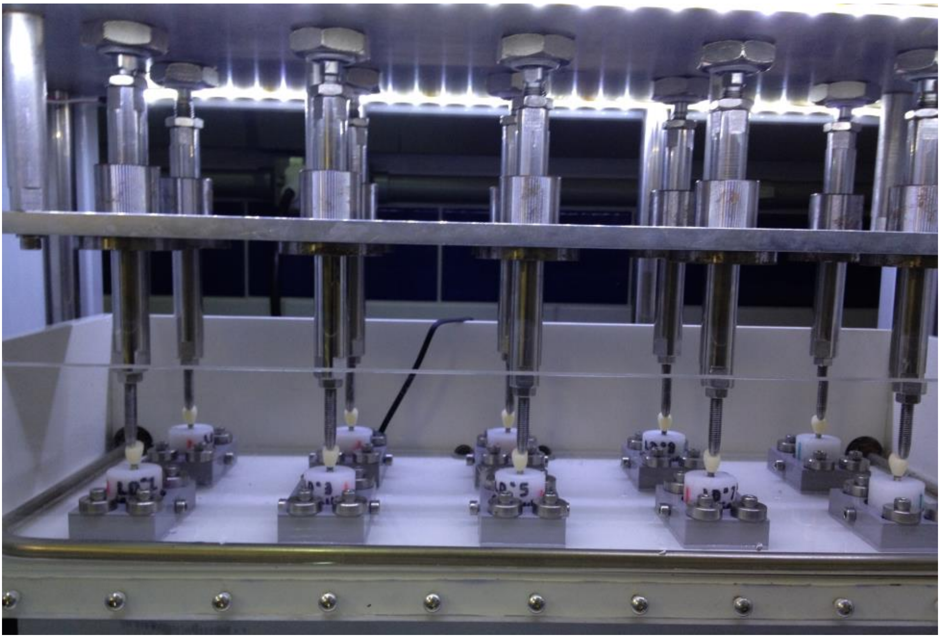


Figure 3.2. Dynamic load fatigue test setting before immersion in artificial chewing simulation.

3.5 *Microscopic characterization of the implant-supported prostheses*

Twelve crown-abutment-implant systems (*n* = 3) were embedded in epoxy resin (Technovit 400, Heraus Kuzer, Germany) and then cross-sectioned at 90 degrees relative to the plane of the implant-abutment joint. The cross-sectioned samples of ceramic crowns and abutment-implant connections were inspected using a Scanning Electron Microscope (SEM Hitachi TM3030, Japan) to evaluate the mechanical integrity of interfaces and to measure microgaps sizes existing between abutment and implant before and after fatigue tests.

3.6 *Statistical analysis*

Data was analyzed using statistic software (Origin 9.1). The Shapiro–Wilk test was first applied to test the assumption of normality. Differences in removal torque between the different experimental groups were compared using 1-way ANOVA followed by Tukey HSD multiple comparison test. P values lower than 0.05 were considered statistically significant in the analysis of the results.

4. RESULTS

4.1. *Removal torque evaluation*

The removal torque mean values of implant-abutment joints are shown in Fig 4.1.

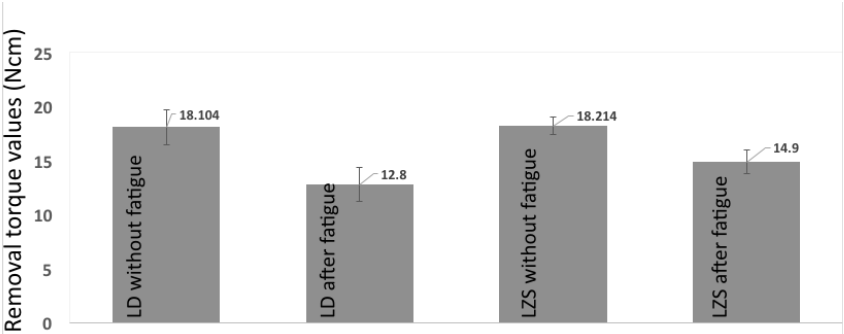


Fig 4.1. Mean removal torque values lithium disilicate (LD) and Zirconium lithium silicate (LZS) subjected or not to fatigue test.

Removal torque mean values for abutments without fatigue and immersed in the Ringer's solutions for 72 h were lower than those for insertion torque on abutments. The removal torque values for LD (18.0 ± 1.6 N.cm) and LZS (18.2 ± 0.8 N.cm) group abutments were around 90% of the insertion torque (20 N.cm), without influence of fatigue.

However, mean values of removal torque on abutments were significantly lower for both LD (12.8 ± 1.6 Ncm) and LZS (14.9 ± 1.1 Ncm) after fatigue tests than those recorded without fatigue tests in the Ringer's solutions for 72 h (Fig. 4.1). After fatigue tests, the removal torque values for LD group abutments were around 64% of the insertion torque, while LZS group abutment screws had removal torque values at 74% of the insertion torque. In fact, there was no significant difference between both glass-ceramic groups (LD and LZS) without fatigue testing. The Tukey multiple comparisons indicated significant differences in removal torque mean values between both groups of abutment assemblies on fatigue tests and free of fatigue ($p = 0.04$).

4.2. ***Microscopic analysis of abutment surfaces and crown-adhesive-abutment interfaces***

The scanning electron microscope (SEM) was used to evaluate surface of abutment before and after bonding/cementation treatment. The surface treatment protocol used sand blasting, hydrofluoric acid-etch, followed by steam cleaning and silane treatment.
SEM images of abutment surfaces before the placement of crowns are shown in Figure 4.2.

In fact, the abutment surface treatment improved the morphological aspect of abutment surfaces that can increase the mechanical interlocking of the luting composite for the prosthetic crowns. SEM images of the ceramic crowns tested in this study are shown in Figures 4.3-4.6. Lithium disilicate (LD) crowns revealed a dense and crystallized structure with a low volume of pores (Figures 4.3A-D and 4.4A-D). On the other hand, the lithium-zirconium silicate glass-ceramics (LZS) displayed more variability in crystalline density and overall porosity (Figures 4.5A-D and 4.6A-D). However, no signs of ceramic fracture were detected on both glass-ceramic materials after fatigue tests (Figures 4.5 and 4.6 A-D).

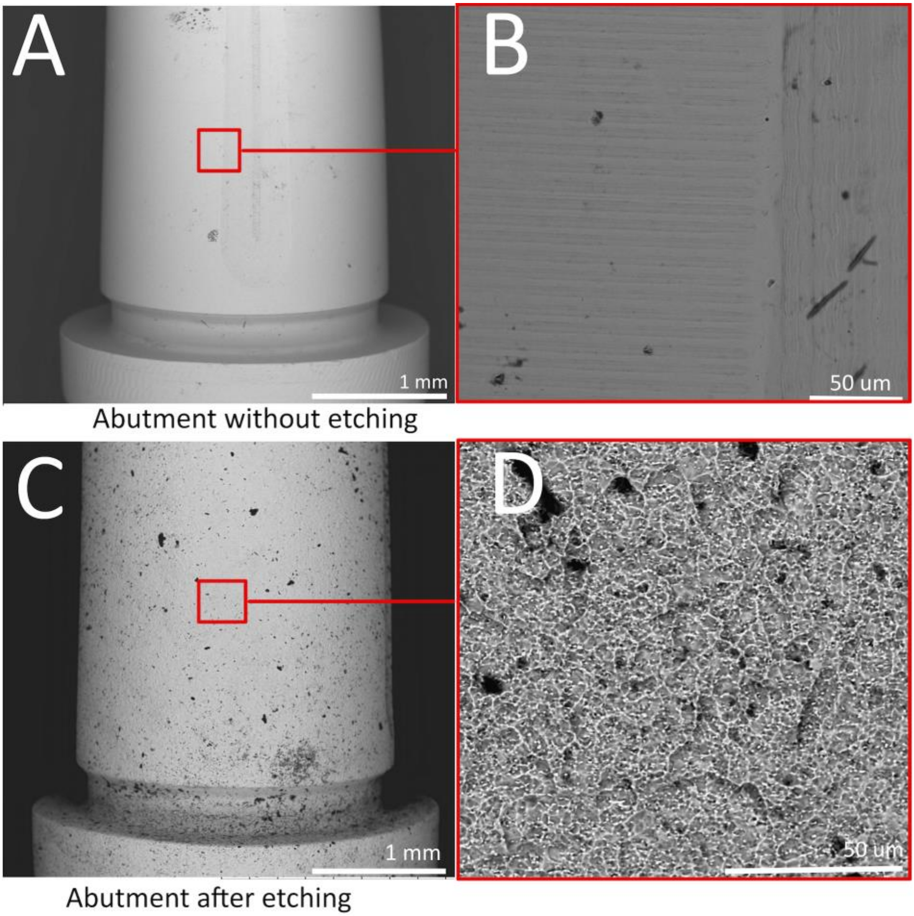


Figure 4.2. SEM images of titanium alloy abutment surface without surface treatment (A,B) and following surface treatment protocol (C,D).

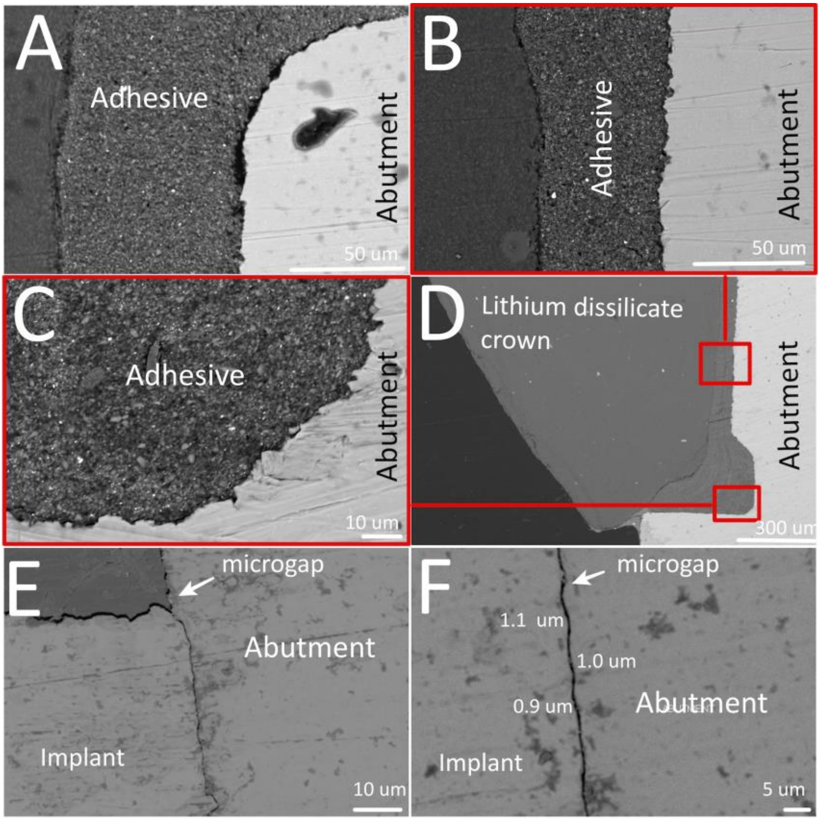


Figure 4.3. Lithium disilicate (LD) crown microscropic evaluation without fatigue testing the crown-cement-abutment interfaces (A-D). E-F show implant-abutment microgap values before fatigue testing.

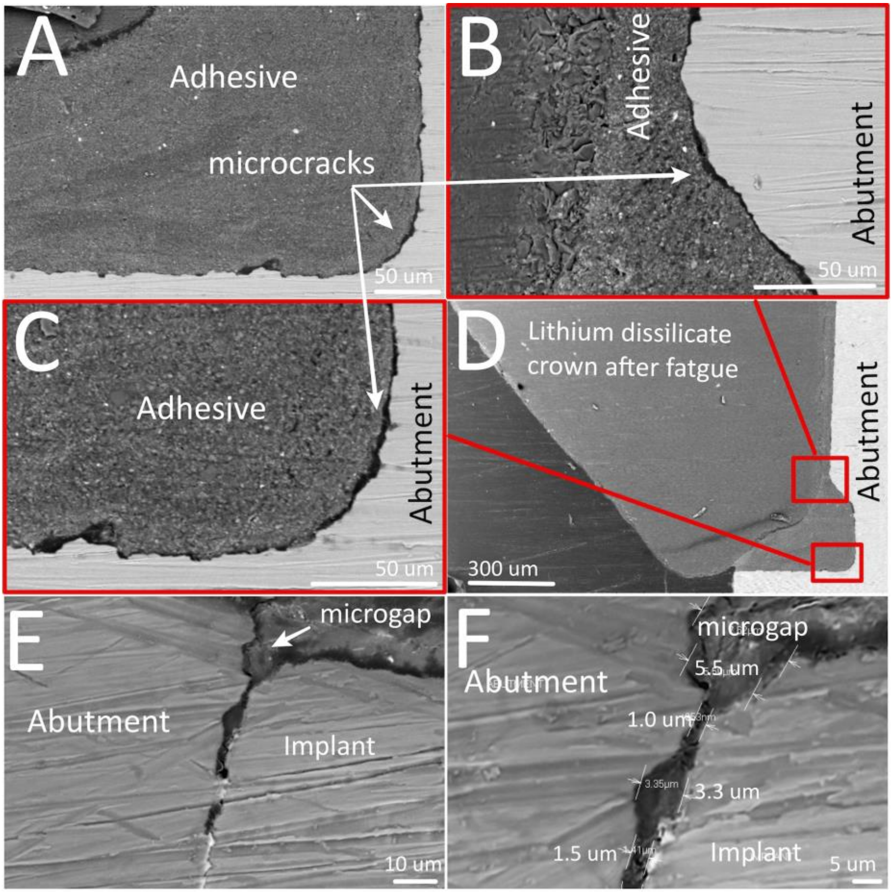


Figure 4.4. Lithium disilicate (LD) crown microscropic evaluation without fatigue testing of crown-cement-abutment interfaces (A-D). Images E-F illustrate implant-abutment microgap values following fatigue testing.

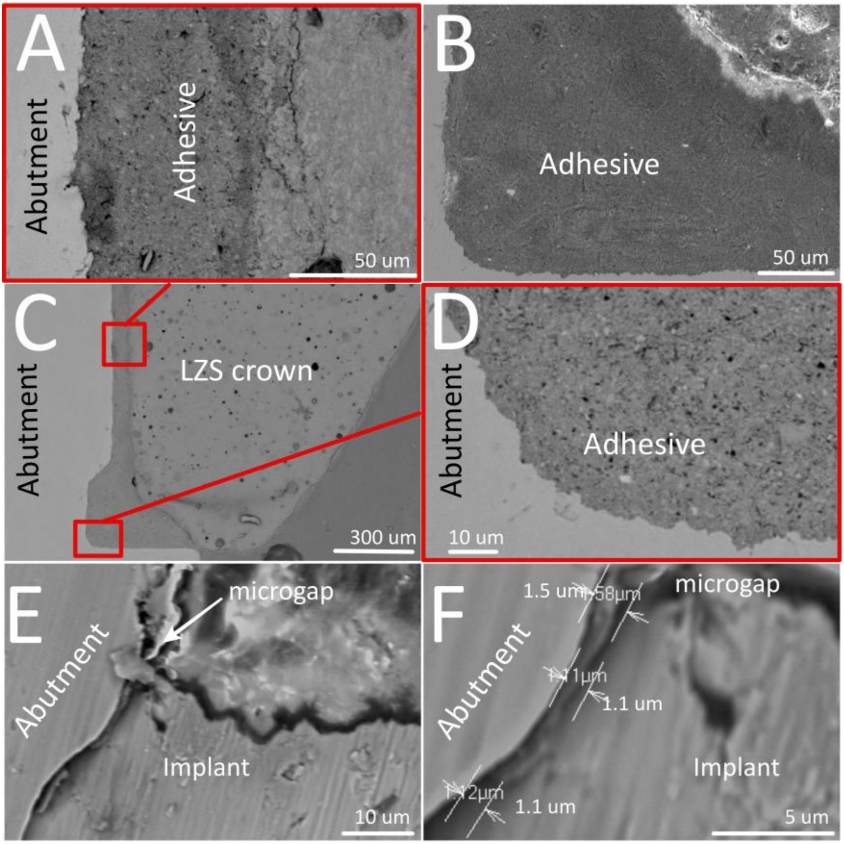


Figure 4.5. Zirconium lithium silicate (LZS) crown microscropic evaluation without fatigue testing the crown-cement-abutment interfaces (A-D). Image E-F illustrating implant-abutment microgap values before fatigue testing.

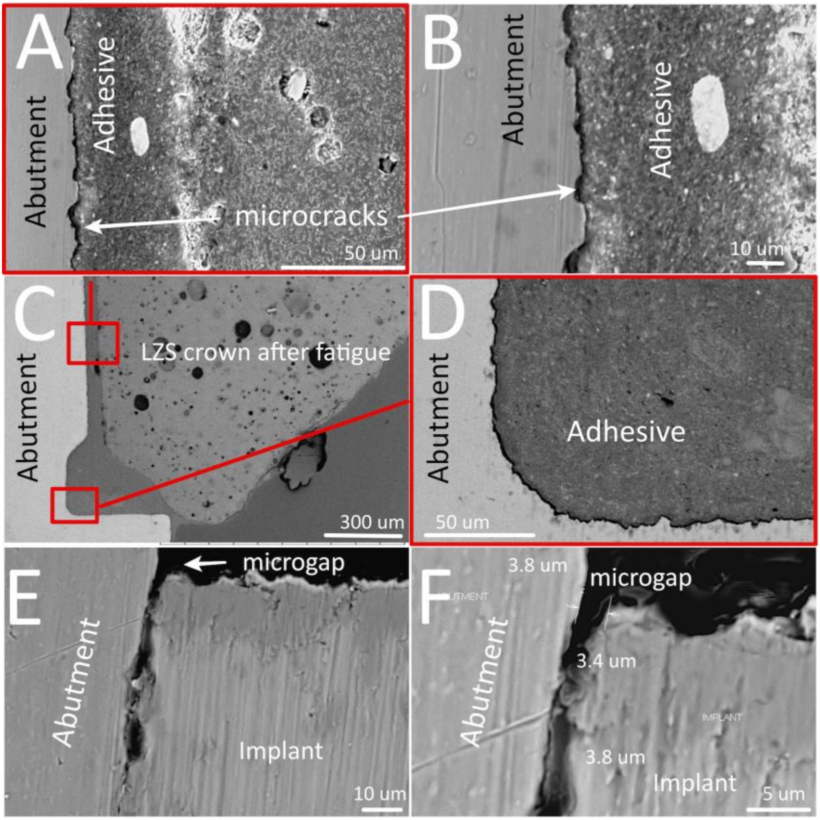


Figure 4.6. Zirconium lithium silicate (LZS) crown SEM evaluation following fatigue test of crown-cement-abutment interfaces (A-D). Images E-F illustrating implant-abutment microgap values following fatigue testing.

4.3. Measurement of microgap sizes between implant and abutment

SEM images of the microgaps at implant-abutment joints for LZS or LD groups free or on fatigue tests are shown in Figures 4.3-4.6. At LD groups, the mean values of microgap size were lower without fatigue tests (1.05 ± 0.2 μm) (Fig. 4.3E and F) than those recorded after fatigue tests (3.8 ± 0.9 μm) (Fig. 4.4E and F). The mean values of microgap size recorded for the LZS group were about 1.1 ± 0.3 μm free of fatigue tests (Fig. 4.5E and F) and 4.4 ± 0.8 μm on fatigue tests (Fig. 4.6E and F).

5. DISCUSSION

The results of the present study support the rejection of the null hypothesis. They showed significant differences in removal torque values recorded for abutments after fatigue tests when compared to abutments free of fatigue ($p < 0.05$). The size of microgaps existing between implant and abutment increased after fatigue tests, as shown by scanning electron microscopy ($p < 0.05$). Also, the propagation of cracks at the crown-adhesive-abutment interfaces was detected by such microscopic analyses. However, no failures were noticed for both lithium disilicate or lithium-zirconium silicate glass-ceramics crowns tested in this study. In fact, the structural materials for crowns and therefore the procedure involving surface treatment of abutment, adhesive bonding and screwing of the crown provided a proper stabilization of the crown avoiding micro-movements through the crown-abutment assembly.

The statistical analysis of this study compared the torque values of Morse taper joint implant-abutment joints immersed in a simulated body solution for 72 h, on and free of fatigue stimuli. Previous studies have reported a decrease of removal torque on implant-abutment joints after fatigue testing despite the test parameters might vary considering the load magnitude and direction, frequency, number of cycles or electrolyte [44-47]. Several variables might cause the loosening of removal torque such as: the design of the implant-abutment joint; the misfit among abutment, abutment screw and implant; surface morphology; magnitude and direction of the occlusal overload; number and position of dental implants; cantilever length; tightening torque; physical properties of materials; oral environment [44-51]. However, there was no significant difference noted between the two all-ceramic prosthetic groups in the removal torque values.

In the present study, the decrease in removal torque values that occured following fatigue testing was further supported by SEM images, noting a widening of the microgap size between implant and abutment. Previous studies have reported the presence of microgaps at different dental implant joints with dimensions ranging from 1.7 up to 10 µm [44,58-60]. As a result of occlusal loading during mastication, micro-movements occur between implant and abutment contacting surfaces resulting in material loss and screw loosening leading to an increase in microgap sizes [44, 61].

It should be highlighted in this study that the abutment surface was treated with grit-blasting procedure and acidic etching to enhance the mechanical interlocking of the luting composite for crown attachment. Considering surface analyses, it is evident that commercial processing of titanium abutment lacks morphological aspects of the surfaces for proper bond strength of the prosthetic-abutment interface. Considering the microscopic results found in the present study, the authors recommend an improvement of the bonding protocol of cement-retained crown to abutments in order to long-term bond strength of the abutment-adhesive-abutment interface. Such improvement can be achieved by increasing the roughness of the abutment surface. Nevertheless, such findings should be evaluated further by complementary *in vitro* evidence-based statistical analysis.

The microscopic evaluation by SEM revealed a significant presence of pores on the microstructure of zirconia-reinforced glass ceramics when compared to that seen on lithium disilicate glass-ceramic microstructure. The densification of glass-ceramics is usually accompanied with an increment of strength and fracture toughness. Even though the SEM images did not confirm a qualitative observable difference in crack propagation between the prosthetic groups, those pores are likely considered flaws during the material processing, that can affect the long term performance of the material in the oral cavity [62]. However, no failure by ceramic fracture was detected after fatigue testing. In materials with comparable crystalline composition such as the test materials, porosity, grain size, shape and orientation of the crystals are important to determine the mechanical integrity of the prosthetic materials [40]. Moreover, variables such as fatigue, cementation, manipulation, design of the restoration and oral health might affect the clinical performance of the glass-ceramics [44,45].

6. CONCLUSIONS

Within the limitations of this study, the main outcomes of this work are as follows:

- Both lithium disilicate and zirconium-lithium silicates glass-ceramics supported by Morse taper implant-abutment joints can be considered successful and effective materials considering no fracture was detected after fatigue tests;

- However, cracks were detected at crown-adhesive and at adhesive-abutment interfaces that can negatively affect the mechanical integrity of the implant-supported prosthesis. The improvement of cement-retaining procedures considering bonding agents and surface treatment can increase the long-term performance of screw- and cement-retained prostheses;

- The loosening of the torque on abutment to implant also occurred in two stages: 1) an initial decrease of removal torque before loading in the pre-load stage; and then 2) subsequently on the loading stage. As a result, the size of microgaps significantly increased after fatigue what can promote the instability of the abutment-implant joint on loading.

7. PERSPECTIVES

The prosthetic crown design and fabrication using modern, novel materials is an ongoing process of improvement. More recent developments using novel composites such as PEEK reinforced with micro- and nano-fillers based on short fibers or irregular particles have results in some promising outcomes [21,35]. Also, the functionally graded materials (FGM) strategy to produce composites and ceramic materials has shown an increase of mechanical performance of the structural materials avoiding abrupt failures commonly found at interfaces [63]. In parallel to this approach a new concept, named *bioinspired materials design,* has recently gained considerable interest mainly due to the growth of interdisciplinary interactions between biologists, chemists, physicists and materials scientists [64,65]. Most of the solutions are based in a bio-inspired design of dental multilayers, trying to approach to the functionally graded structures of the dentin enamel-junction (DEJ) in natural teeth. Understanding this concept, a new research area starts to develop several multilayer dental systems for dental restoration. Some authors propose a graded transition between porcelain and other materials, low-modulus glass, lithium disilicate or zirconia, to decrease shipping effects [62-68].

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